[Document Name] CLAIMS

[Claim 1]

A packaged beverage containing from 0.06 to 0.5 wt% of non-polymer catechins, wherein the packaged beverage has been obtained by incorporating a tea extract obtained from tea leaves of the genus Camellia subjected beforehand to a treatment of contacting with carbon dioxide in a supercritical state.

[Claim 2]

The packaged beverage according to claim 1, wherein the tea leaves are non-fermented tea leaves.

[Claim 3]

The packaged beverage according to claim 1 or 2, wherein the weight ratio of the non-polymer catechins to caffeine in the packaged beverage is 10 or greater.

[Claim 4]

The packaged beverage according to any one of claims 1-3, which is a packaged non-tea beverage.

[Claim 5]

A process for production of a packaged beverage containing from 0.06 to 0.5 wt% of non-polymer catechins, which comprises incorporating a tea extract obtained from tea leaves of the genus Camellia subjected beforehand to a treatment of contacting with carbon dioxide in a supercritical state.

[Document Name] SPECIFICATION

[Title of the Invention] PACKAGED BEVERAGE

[Field of the Invention]

[0001]

This invention relates to a packaged beverage, which contains catechins at high concentration, is lowered in green tea favor, and undergoes little in flavor during storage at high temperatures.

[Background of the Invention]

[0002]

As effects of catechins,  $\alpha$ -amylase activity inhibiting effect and the like have been reported (see, for example, Patent Document 1). To develop such physiological effects, it is necessary for an adult to drink 4 to 5 cups of tea per day. Accordingly, there is a desire for a technology that permits addition of catechins at a high concentration to beverages to facilitate ingestion of a large amount of catechins. As one of such methods, there is a method that adds catechins in a dissolved state to beverages by using a concentrate of a green tea extract (see, for example, Patent Document 2) or the like.

[Patent Document 1] JP-A-03-133928

[Patent Document 2] JP-A-59-219384

[Disclosure of the Invention]

[Problem to Be Solved by the Invention]

[0003]

However, a high-catechin beverage in which a concentrate of a green tea extract is incorporated has hardly been formulated into the form of another beverage or drink, for example, an isotonic drink or the like, because it has not only bitterness but also a flavor typical to green tea. Further, a packaged beverage in which a concentrate of a green tea extract is incorporated has been found to develop a problem that by storage at high temperatures, its flavor changes to a green-tea-derived flavor which was not sensed shortly after the incorporation.

An object of the present invention is, therefore, to provide a packaged beverage, which contains at high concentration catechins having physiological effects, is lowered in green tea favor, and does not undergo much change in flavor even when stored at high temperatures.

[Means for Solving the Problem]

[0004]

The present inventors have, therefore, conducted an investigation about a method for extracting non-polymer catechins from tea leaves. As a result, it has been found that non-polymer catechins are selectively extracted when extraction is conducted using tea leaves subjected beforehand to a treatment of contacting with carbon dioxide in a supercritical state, i.e., a residue of the treatment, that

a packaged beverage with the tea extract incorporated therein is lowered in tea flavor, and that even when stored at high temperatures, the packaged beverage does not develop a green-tea-derived flavor which is not sensed shortly after the incorporation, and as a consequence, is free from a change in flavor.

[0005]

Described specifically, the present invention provides a packaged beverage containing from 0.06 to 0.5 wt% of non-polymer catechins, wherein said packaged beverage has been obtained by incorporating a tea extract extracted from tea leaves of the genus Camellia subjected beforehand to a treatment of contacting with carbon dioxide in a supercritical state; and also its production process.

[Advantageous Effects of the Invention]

[0006]

The packaged beverage according to the present invention contains at high concentration catechins having physiological effect, undergoes no change in flavor even when stored at high temperatures, and is lowered in tea favor; and therefore, is useful a so-called non-tea beverage such as an isotonic drink, a sports drink, a vegetable-juice-containing beverage or a fruit-juice-containing beverage.

[Preferred Embodiments of the Invention]

[0007]

Incorporated in the packaged beverage according to the present invention is an extract obtained from tea leaves belonging to the Genus Camellia and subjected beforehand to a treatment of contacting with carbon dioxide in a supercritical state. The tea leaves for use in the present invention can be either raw tea leaves or manufactured tea leaves insofar as they belong to the Genus Camellia. As the manufactured tea leaves, non-fermented tea is more preferred. Preferred examples of steamed tea leaves include sencha (middle-grade green tea), fukamushicha (deep-steamed green tea), gyokuro (shaded green tea), kabusecha (partially shaded green tea), mushi-tamaryokucha (steamed, rounded, beads-shaped green tea), and bancha (coarse green tea). Preferred examples of roasted tea leaves, on the other hand, include kamairi-tamaryokucha (roasted, rounded, beads-shaped green tea) and Chinese green tea. As manufactured tea leaves, steamed tea leaves or dipped tea leaves are preferred from the standpoint of avoiding generation of an additional flavor and taste derived from tea leaves during roasting.

[8000]

In the present invention, an extract containing non-polymer catechins is obtained from tea leaves available as a residue in the supercritical extraction. The present invention relates to a beverage with the extract incorporated therein. There have conventionally been several techniques

for obtaining flavor components from tea leaves by supercritical extraction (JP-A-2001-293076, JP-A-10-77496, JP-A-06-133726, and JP-A-06-184591). Any of these techniques uses a supercritical extract of tea leaves, however, and there is no description as to a technique that makes use of residual tea leaves after supercritical extraction or as to the components that remain in the residual tea leaves.

[0009]

In the present invention, a process for production of a tea extract from tea leaves, which belong to the Genus Camellia and have been subjected to a treatment to contact with carbon dioxide in its supercritical state, includes specifically (A) a step of moistening green tea leaves, (B) a step of treating the green tea leaves with carbon dioxide in its supercritical state, (C) a step of extracting a green tea extract from the tea leaves, and (D) a step of purifying the green tea extract as needed depending on the kind of a beverage to be produced. Each of these steps will hereinafter be described.

[0010]

In the step (A), from 0.2 to 0.4 weight part of a 75:25 to 99.5:0.5 mixture of ethanol and water is at first added to 1 weight part of the green tea leaves to moisten the green tea leaves. Without this moistening step of the green tea leaves with the 75:25 to 99.5:0.5 mixture of ethanol and water, elimination of a green tea flavor in the step (B) cannot be

achieved sufficiently. Addition of the 75:25 to 99.5:0.5 mixture of ethanol and water in an amount smaller than 0.2 weight part can bring about no sufficient effect for the elimination of the green tea flavor, and therefore, is not preferred. On the other hand, addition of the mixture in an amount greater than 0.4 weight part leads to a reduction in extraction efficiency, and therefore, is not preferred.

The ratio of ethanol to water in the mixture is preferably from 75:25 to 99.5:0.5, more preferably from 80:20 to 99.5:0.5, still more preferably from 80:20 to 90:10. The mixture of ethanol and water is added in an amount of from 0.2 to 0.4 weight part, preferably from 0.3 to 0.4 weight part.

The green tea leaves to which the above-described predetermined amount of the mixture of ethanol and water has been added is preferably left to stand at from 0 to 100°C for 0.5 hour or longer to have the green tea leaves moistened sufficiently.

[0011]

In the step (B), carbon dioxide in a supercritical state in which a 75:25 to 99.5:0.5 mixture of ethanol and water has been added is brought into contact with the moistened green tea leaves. According to the step (B), the water content of the green tea leaves can be retained constant so that the green tea flavor in the green tea leaves can be efficiently eliminated. On the other hand, the catechins in the green tea remain

substantially completely in the green tea leaves, and are not impaired by the above operation.

[0012]

The ratio of ethanol to water in the mixture to be used is preferably from 75:25 to 99.5:0.5, more preferably from 75:25 to 95:5, still more preferably from 80:20 to 90:10. The mixture of ethanol and water is added in an amount of from 0.02 to 0.04 weight part, preferably from 0.03 to 0.04 weight part per weight part of carbon dioxide.

[0013]

As to the carbon dioxide to be used, elimination of green tea flavor components can be efficiently achieved insofar as carbon dioxide is in a supercritical state (pressure: 7 MPa or higher, temperature: 31°C or higher). From the standpoint of the efficiency of elimination of bitter components and green tea flavor, however, carbon dioxide at from 20 to 50 MPa and at 35 to 100°C, particularly at from 30 to 40 MPa and at 60 to 80°C is preferred. Carbon dioxide can be used in a proportion of preferably 20 weight parts or more, more preferably from 20 to 250 weight parts, still more preferably from 50 to 150 weight parts per weight part of the green tea leaves.

[0014]

Ethanol and/or water and supercritical carbon dioxide are brought into contact with the green tea leaves at the same time. For example, an aqueous ethanol solution and

supercritical carbon dioxide may be brought into contact with the tea leaves at the same time, or an aqueous ethanol solution and supercritical carbon dioxide may be mixed together in advance, and may then be brought into contact with the green tea leaves. To bring ethanol and/or water and supercritical carbon dioxide into contact with the green tea leaves, use of an ordinary supercritical extraction apparatus is preferred. In general, the above-described contact may be effected preferably at a feed rate of from 10 to 25 weight parts/hour of supercritical carbon dioxide per weight part of green tea leaves to retain the water content of the green tea leaves although the feed rate may vary depending on the capacity of the apparatus.

[0015]

In the step (C), the extraction from the green tea leaves which have been brought into contact with the supercritical carbon dioxide is conducted by using from 10 to 150 weight parts of water per weight part of the green tea leaves. To maximize the efficiency of extraction of water-soluble components led by catechins, water may be used preferably in an amount of from 20 to 100 weight parts, more preferably in an amount of from 20 to 50 weight parts.

[0016]

The extraction in the step (C) can be conducted under usual extraction conditions. Upon conducting the extraction

from the green tea leaves, the temperature can be changed as needed depending on the kind of the tealeaves. The temperature is preferably, for example, from 60 to 90°C in the case of sencha (middle-grade green tea) or gyokurocha (shaded green tea), or from 50 to 60°C or so in the case of gyokuro (shaded green tea) or kabusecha (partially shaded green tea). In the case of bancha (coarse tea), on the other hand, a temperature of from 90°C to boiling water can be used. The time of extraction from the green tea leaves is preferably from 1 to 60 minutes, more preferably from 1 to 40 minutes, still more preferably from 1 to 30 minutes. In the case of a process for production of an extract from middle-grade sencha leaves, for example, an extract can be obtained by adding the tea leaves into ion-exchanged water heated at 65°C, stirring them for 2 minutes or so, leaving them to stand also for 2 minutes or so, filtering out the green tea leaves, and then filtering out fragmented tea leaves with a flannel filter cloth.

[0017]

As filter media usable in the step (D), activated carbon, acid clay, diatomaceous earth, talc, magnesium oxide, kaolin, bentonite, perlite, silica gel, activated alumina, celite and the like can be mentioned specifically. These filter media can be used either singly or in combination. Among these filter media, preferred are activated carbon and acid clay.

As the activated carbon for use in the present invention,

any activated carbon can be used insofar as it is generally used on an industrial level. Usable examples include commercially-available products such as "ZN-50" (product of Hokuetsu Carbon Industry Co., Ltd.), "KURARAY COAL GLC", "KURARAY COAL PK-D" and "KURARAY COAL PW-D" (products of Kuraray Chemical K.K.), and "SHIROWASHI AW50", "SHIROWASHI A", "SHIROWASHI M" and "SHIROWASHI C" (products of Takeda Pharmaceutical Company Limited).

The pore volume of the activated carbon is preferably from 0.01 to 0.8 mL/g, more preferably from 0.1 to 0.7 mL/g. The activated carbon having a specific surface area in a range of from 800 to 1,300 m $^2$ /g, particularly from 900 to 1,200 m $^2$ /g is preferred. It is to be noted that these physical values are those determined by the nitrogen adsorption method.

## [0018]

The activated carbon can be added preferably in a proportion of from 0.5 to 5 weight parts, particularly in a proportion of from 0.5 to 3 weight parts to 100 weight parts of the mixture of the organic solvent and water. The addition of activated carbon in an excessively small proportion leads to deteriorated elimination efficiency of bitterness components, while the addition of activated carbon in an unduly large proportion results in higher cake resistance in the filtration step. Proportions outside the above-described range are hence not preferred.

[0019]

The treatment of the green tea extract to contact it with activated carbon can be conducted by a batchwise treatment method, a continuous treatment method making use of a column, or the like. Preferred in general is a method in which powdery activated carbon is added, the resulting mixture is stirred to selectively adsorb bitterness components, and a filtering operation is then conducted to obtain a filtrate free of bitterness components, or a method in which bitterness components are selectively adsorbed by a continuous treatment using a column packed with granular activated carbon.

Acid clay and activated clay for use in the present invention both contain, as general chemical components,  $SiO_2$ ,  $Al_2O_3$ ,  $Fe_2O_3$ , CaO, MgO, etc., and those having a  $SiO_2/Al_2O_3$  ratio of from 3 to 12, particularly from 4 to 9 are preferred. Also preferred are those of compositions which contain from 2 to 5 wt% of  $Fe_2O_3$ , from 0 to 1.5 wt% of CaO and from 1 to 7 wt% of MgO.

Activated clay is obtained by treating a naturally-occurring acid clay (montmorillonite clay) with a mineral acid such as sulfuric acid.

[0020]

The specific surface area of acid clay or activated clay is preferably from 50 to 350  $\text{m}^2/\text{g}$  although it varies depending on the degree of the acid treatment or the like, and its pH

(5% suspension) is preferably from 2.5 to 8, particularly from 3.6 to 7. Usable examples of acid clay include commercially-available products such as "MIZUKA ACE #600" (product of Mizusawa Chemical Industries, Ltd.).

[0021]

Acid clay or activated clay can be added preferably in a proportion of from 2.5 to 25 weight parts, particularly in a proportion of from 2.5 to 15 weight parts to 100 weight parts of the mixture of the organic solvent and water. The addition of acid clay or activated clay in an unduly small proportion leads to a deterioration in elimination efficiency of bitterness components, while the addition of acid clay or activated clay in an excessively large proportion leads to an increase in the cake resistance in the filtration step. It is, therefore, not preferred to add acid clay or activated clay in a proportion outside the above-described range.

[0022]

The weight ratio of activated carbon to acid clay or activated clay can be from 1 to 10 of acid clay or activated clay to 1 of activated carbon, with the weight ratio of activated carbon: acid clay or activated clay = 1:1 to 1:6 being preferred.

Upon bringing activated carbon and acid clay or activated clay into contact with the green tea extract, they may be both brought into contact with the green tea extract at the same time, or may be brought into contact separately one after the

other (in an arbitrary order).

[0023]

The treatment of the green tea extract to contact it with activated carbon and acid clay or activated clay can be conducted by a batchwise treatment method, a continuous treatment method making use of a column, or the like. Adopted in general is a method in which powdery activated carbon or the like is added, the resulting mixture is stirred to selectively adsorb bitterness components, and a filtering operation is then conducted to obtain a filtrate free of bitterness components, or a method in which bitterness components are selectively adsorbed by a continuous treatment using a column packed with granular activated carbon or the like. Further, the form of the green tea extract can be either a liquid form or a solid form.

[0024]

From the solution containing the green tea extract, the organic solvent can be distilled away by using a method such as reduced-pressure distillation. To prepare the green tea extract into a solid form, it may be formed into powder by a method such as freeze drying or spray drying.

It is important from the standpoint of the effect of lowering a green tea flavor that the total amount of terpene alcohols, aliphatic oxides and aromatic alcohols in the resulting green tea extract is less than 30 wt% of their total

amount in the case of a water extract of green tea.

It is preferred from the standpoint of the flavor of a beverage that in terms of the total amount of linalol oxide, phenethyl alcohol and geraniol or in terms of linalol oxide (cis, furanoid), for example, their total amount in the tea extract may be less than 30 wt%, preferably 25 wt% or lower, more preferably 20 wt% or lower when their content in a water extract of green tea is assumed to be 100.

[0025]

As a result of the supercritical extraction treatment, the thus-obtained green tea extract, despite the inclusion of catechins at high concentrations, is lowered in the green teaflavor, so that it can be used as a material for high-catechin isotonic drinks, vegetable-juice-containing beverages, fruit-juice-containing beverages, sports drinks, and the like.

The non-polymer catechins/caffeine weight ratio of the resultant green tea extract may be preferably from 10 to 1000, more preferably from 40 to 1000, still more preferably from 40 to 500. An unduly low weight ratio leads to the impartment of bitterness derived from impurities in the green tea extract upon its incorporation in a beverage, while an excessively high weight ratio results in higher purification cost.

[0026]

The packaged beverage according to the present invention

contains non-polymer catechins dissolved in water in an amount of from 0.06 to 0.5 wt%, preferably from 0.08 to 0.5 wt%, more preferably from 0.092 to 0.4 wt%, still more preferably from 0.1 to 0.3 wt%. A content of non-polymer catechins in the above-described range is preferred in that a great deal of non-polymer catechins can be taken with ease. The concentration of the non-polymer catechins can be adjusted by the amount of the green tea extract to be incorporated.

Further, the daily intake required for an adult to exhibit the effects of the promotion of accumulated fat burning, the promotion of dietary fat burning and the promotion of  $\beta$ -oxidation gene expression in the liver is preferably 300 mg or more, more preferably 450 mg or more, still more preferably 500 mg or more in terms of non-polymer catechins. Specifically, it has been confirmed that an anti-puffiness effect and/or visceral fat reducing effect can be brought about by ingesting a beverage which contains 483 mg, 555 mg, 900 mg or the like of non-polymer catechins per package (JP-A-2002-326932).

Therefore, the daily intake of the packaged beverage according to the present invention for an adult can also be preferably 300 mg or more, more preferably 450 mg or more, still more preferably 500 mg or more in terms of non-polymer catechins. From the standpoint of assuring the required daily intake amount, the non-polymer catechins may be contained in an amount of preferably 300 mg or more, more preferably 450

mg or more, still more preferably 500 mg or more per package (350 mL to 500 mL) of the packaged beverage according to the present invention.

[0027]

The term "non-polymer catechins" as used herein is a generic term which collectively encompasses non-epicatechins such as catechin, gallocatechin, catechin gallate and gallocatechin gallate, and epicatechins such as epicatechin, epigallocatechin, epicatechin gallate and epigallocatechin gallate.

The weight ratio of non-polymer catechins to caffeine in the package beverage in which the resultant green tea extract has been incorporated may be preferably 10 or greater, more preferably from 10 to 1000, still more preferably from 40 to 1000, even more preferably from 40 to 500. An unduly low weight ratio leads to the impartment of bitterness derived from impurities in the green tea extract upon its incorporation in a beverage, while an excessively high weight ratio results in higher purification cost.

[0028]

In the packaged beverage according to the present invention, a sweetener may also be used to improve the taste. As the sweetener, an artificial sweetener, carbohydrate or glycerol can be used. The content of such a sweetener in the packaged beverage according to the present invention is

preferably from 0.0001 to 20 wt%, more preferably from 0.001 to 15 wt%, still more preferably from 0.001 to 10 wt%. A content lower than 0.0001 wt% cannot impart much sweetness and cannot achieve a balance between sourness and saltiness, while a content higher than 20 wt% leads to excessive sweetness and therefore, to the strong feeling of being caught in the throat and a reduction in the feeling as the beverage passes down the throat.

[0029]

Among such sweeteners, example of the artificial sweetener usable in the present invention include high-sweetness sweeteners such as saccharin, saccharin sodium, aspartame, sucralose and neotame; and sugar alcohols such as sorbitol, erythritol and xylitol. Specifically, artificial sweeteners, that is, "ACESULFAME-K", "SLIM-UP SUGAR", "LAKANTO-S", "PALSWEET" etc. can be used as needed. As a combination of sweeteners, a combination of artificial sweetener alone or a combination of an artificial sweetener and a fructose compound is more preferred. The content of such an artificial sweetener may be from 0.0001 to 20 wt%.

[0030]

As a carbohydrate, a soluble carbohydrate is used. The carbohydrate can be a mixture of glucose and fructose, or a carbohydrate hydrolyzable into glucose and fructose or capable of forming glucose and fructose in the digestive tract. The

term "carbohydrate" as used herein includes monosaccharides, oligosaccharides, conjugated polysaccharides, and mixtures thereof.

[0031]

Monosaccharides include tetroses, pentoses, hexoses and ketohexoses. Examples of the hexoses are aldohexoses such as glucose known as grape sugar. The content of glucose in the packaged beverage according to the present invention can be preferably from 0.0001 to 20 wt%, more preferably from 0.001 to 15 wt%, still more preferably from 0.001 to 10 wt%. The content of fructose in the packaged beverage according to the present invention can be preferably from 0.0001 to 20 wt%, more preferably from 0.0001 to 10 wt%.

[0032]

As oligosaccharides out of the sweeteners usable in the packaged beverage according to the present invention, sucrose, maltodextrin, corn syrup, and fructose-rich corn syrup can be mentioned. An illustrative disaccharide is sucrose known as cane sugar or beet sugar. The content of sucrose in the packaged beverage according to the present invention can be preferably from 0.001 to 20 wt%, more preferably from 0.001 to 15 wt%, particularly preferably from 0.001 to 10 wt%.

A glycerol can be used at from 0.1 to 15 wt%, preferably from 0.1 to 10 wt% in the packaged beverage according to the

present invention.

[0033]

A preferred example of the conjugated polysaccharides as sweeteners usable in the present invention is maltodextrin.

[0034]

The total carbohydrate usable in the packaged beverage according to the present invention can be from 0.0001 to 20 wt% of the total weight. For example, the total amount of carbohydrates includes not only those naturally existing in a fruit juice or a tea extract but also added carbohydrate(s). The carbohydrates include, for example, carbohydrate derivatives, polyols, artificial sweeteners, and the like.

[0035]

In the packaged beverage according to the present invention, sodium ions and potassium ions may be contained at from 0.001 to 0.5 wt% and from 0.001 to 0.2 wt%, respectively.

[0036]

As sodium ions, a readily-available sodium salt such as sodium chloride, sodium carbonate, sodium hydrogencarbonate, sodium citrate, sodium phosphate, sodium hydrogenphosphate, sodium tartrate, sodium benzoate or a mixture thereof can be added, and those derived from an added fruit juice or tea components are also included. Alower sodium ion concentration is desired from the standpoint of facilitating the absorption of water owing to osmotic pressure.

It is, however, important in the present invention that the sodium ion concentration is at such a level as to avoid suction of water into the intestine from the body by osmotic pressure. The sodium ion concentration required to achieve such a level can preferably be lower than the sodium ion concentration in the plasma. The content of sodium ions in the packaged beverage according to the present invention can be preferably from 0.001 to 0.5 wt%, more preferably from 0.002 to 0.4 wt%, particularly from 0.003 to 0.2 wt%.

[0037]

As potassium ions, a potassium salt such as potassium chloride, potassium carbonate, potassium sulfate, potassium acetate, potassium hydrogencarbonate, potassium citrate, potassium phosphate, potassium hydrogenphosphate, potassium tartrate, potassium sorbate or a mixture thereof can be added, and those derived from an added fruit juice or tea components are also included. The content of potassium ions in the packaged beverage according to the present invention can be preferably from 0.001 to 0.2 wt%, more preferably from 0.002 to 0.15 wt%, particularly from 0.003 to 0.12 wt%.

[0038]

In addition to sodium ions and potassium ions, from 0.001 to 0.5 wt%, preferably from 0.002 to 0.4 wt%, still more preferably from 0.003 to 0.3 wt% of chloride ions can also be incorporated in the beverage according to the present

invention. A chloride ion ingredient can be added in the form of a salt such as sodium chloride or potassium chloride. Trace ions such as calcium, magnesium, zinc and/or iron ions may also be added. These ions can also be added in the form of salt or salts. The total amount of existing ions includes not only an amount of ions added but also an amount of ions naturally existing in the beverage. When sodium chloride is added, for example, the amounts of sodium ions and chloride ions in the added sodium chloride are also included in the total amount of ions correspondingly.

[0039]

Addition of a bitterness suppressor to the packaged beverage according to the present invention facilitates its drinking, and therefore, is preferred. As a bitterness suppressor to be used, cyclodextrin is preferred. As the cyclodextrin, an  $\alpha$ -,  $\beta$ - or  $\gamma$ -cyclodextrin or a branched  $\alpha$ -,  $\beta$ - or  $\gamma$ -cyclodextrin or a branched  $\alpha$ -,  $\beta$ - or  $\gamma$ -cyclodextrin can be used. In the beverage, a cyclodextrin may be contained preferably in an amount of from 0.005 to 0.5 wt%, more preferably from 0.01 to 0.3 wt%. To the packaged beverage according to the present invention, it is possible to add as ingredient or ingredients usable in accordance with a formula in addition to the components derived from tea, either singly or in combination, additives such as antioxidants, flavorings, various esters, organic acids, organic acid salts, inorganic acids, inorganic acid salts,

inorganic salts, dyes, emulsifiers, preservatives, seasoning agents, sweeteners, sour seasonings, gums, emulsifiers, oils, vitamins, amino acids, fruit extracts, vegetable extracts, flower honey extracts, pH regulators and quality stabilizers.

[0040]

To the beverage according to the present invention, one or more of flavorings and fruit juices may preferably be added to improve the taste. In general, the juice of a fruit is called "fruit juice" and a flavoring is called "flavor". Natural or synthetic flavorings and fruit juices can be used in the present invention. They can be selected from fruit juices, fruit flavors, plant flavors, and mixtures thereof. For example, a combination of a fruit juice with a tea flavor, preferably a green tea or black tea flavor provides a preferable taste. Preferred fruit juices include apple, pear, lemon, lime, mandarin, grapefruit, cranberry, orange, strawberry, grape, kiwi, pineapple, passion fruit, mango, guava, raspberry and cherry juices. More preferred are citrus juices, specifically grapefruit, orange, lemon, lime and mandarin juices, mango juice, passion fruit juice, quava juice, and mixtures thereof. Preferred natural flavors include jasmine, chamomile, rose, peppermint, Crataegus cuneata, chrysanthemum, water caltrop, sugarcane, bracket fungus of the genus Fomes (Fomes japonicus), bamboo shoot, and the like. Such a juice can be contained preferably in an amount of from

0.001 to 20 wt%, more preferably from 0.002 to 10 wt% in the beverage according to the present invention. Fruit flavors, plant flavors, tea flavors and mixtures thereof can also be used as flavorings. Particularly preferred flavorings are citrus flavors including orange flavor, lemon flavor, lime flavor and grapefruit flavor. As other fruit flavors, apple flavor, grape flavor, raspberry flavor, cranberry flavor, cherry flavor, pineapple flavor and the like are usable. These flavorings can be derived from natural sources such as fruit juices and balms, or can be synthesized. The term "flavoring" as used herein can also include blends of various flavors, for example, a blend of lemon and lime flavors and blends of citrus flavors and selected spices (typically, cola or soft drink flavor). As flavorings of hydrophobic concentrates or extracts, synthetic flavor esters, alcohols, aldehydes, terpenes, sesquiterpenes and the like can be added. Such a flavoring can be added preferably in an amount of from 0.0001 to 5 wt%, more preferably from 0.001 to 3 wt% to the beverage according to the present invention.

## [0041]

The beverage according to the present invention may also contain a sour seasoning as needed. A sour seasoning can be used to maintain the pH of the beverage according to the present invention within from 2 to 6. The acid can be used either in a non-dissociated form or in the form of its potassium salt

or potassium salt. Preferred acids can be edible organic acids including citric acid, malic acid, fumaric acid, adipic acid, phosphoric acid, gluconic acid, tartaric acid, ascorbic acid, acetic acid, phosphoric acid, and mixtures thereof. Most preferred acids are citric acid and malic acid. These sour seasonings are also useful as antioxidants which stabilize the ingredients in the beverage. Examples of other antioxidants include ascorbic acid, plant extracts, and the like.

### [0042]

In the beverage according to the present invention, one or more vitamins can be incorporated further. As preferred vitamins, vitamin A, vitamin C and vitamin E can be mentioned. Other vitamins such as vitamin D and vitamin B can also be used. Minerals can also be used in the beverage according to the present invention. Preferred minerals include calcium, chromium, copper, fluorine, iodine, iron, magnesium, manganese, phosphorus, selenium, silicon, molybdenum, and zinc. Particularly preferred minerals are magnesium, phosphorus, and iron.

### [0043]

As the packaged beverage according to the present invention, a tea beverage or non-tea beverage can be mentioned. Tea beverages include green tea, oolong tea, black tea, blend tea, and the like. As non-tea beverages, beverage forms such

as sports drinks and isotonic beverages are feasible. The term "sports drink" is generally defined to mean a drink that can promptly replenish water and minerals lost in the form of sweat during physical exercise.

As the beverage form in the present invention, a non-tea beverage is preferred.

[0044]

As in general beverages, a package used for the packaged beverage according to the present invention can be provided in a conventional form such as a molded package made essentially of polyethylene terephthalate (a so-called PET bottle), a metal can, a paper container combined with metal foils or plastic films, a bottle or the like. The term "packaged beverage" as used herein means a beverage that can be taken without dilution.

[0045]

The packaged beverage according to the present invention can be produced, for example, by filling the beverage in a package such as a metal can and, when heat sterilization is feasible, conducting heat sterilization under sterilization conditions as prescribed in the Food Sanitation Act. For packages which cannot be subjected to retort sterilization such as PET bottles or paper packages, adopted is a process in which the beverage is sterilized beforehand at a high temperature for a short time under similar sterilization

conditions as those described above, for example, by a plate-type heat exchanger or the like, is cooled to a predetermined temperature, and is then filled in a package. Under aseptic conditions, additional ingredients may be added to and filled in a beverage-filled package. It is also possible to conduct such an operation that subsequent to heat sterilization under acidic conditions, the pH of the beverage is brought back to neutral under aseptic conditions or that subsequent to heat sterilization under neutral conditions, the pH of the beverage is brought back to acidic under aseptic conditions.

[Examples]

[0046]

## Measurement of catechins

A high-performance liquid chromatograph (model: "SCL-10AVP") manufactured by Shimadzu Corporation was fitted with a liquid chromatograph column packed with octadecyl-introduced, "L-Column, TM ODS" (4.6 mm × 250 mm; product of Chemicals Evaluation and Research Institute, Japan). A packaged beverage was filtered through a filter (0.8 µm), diluted with distilled water, and then subjected to chromatography at a column temperature of 35°C by the gradient elution method. A solution A, mobile phase, was a solution containing 0.1 mol/L of acetic acid in distilled water, while a solution B was a solution containing 0.1 mol/L of acetic

acid in acetonitrile. The measurement was conducted under the conditions of 20  $\mu L$  sample injection volume and 280 nm UV detector wavelength.

[0047]

# Measurement of caffeine

## (Analyzer)

A HPLC system (manufactured by Hitachi, Ltd.) was used.

Plotter: "D-2500", Detector: "L-4200", Pump: "L-7100",

Autosampler: "L-7200", Column: "Intersil ODS-2" (2.1

mm in inner diameter  $\times$  250 mm in length).

# (Analytical conditions)

Sample injection volume: 10  $\mu$ L

Flow rate: 1.0 mL/min

Detection wavelength of UV spectrophotometer: 280 nm Eluent A: A 0.1 M solution of acetic acid in water Eluent B: A 0.1 M solution of acetic acid in acetonitrile Concentration gradient conditions (vol. %)

Time	Eluent A	Eluent B
0 min	97%	3%
5 min	97%	3%
37 min	80%	20%
43 min	80%	20%
43.5 min	0%	100%
48.5 min	0%	100%
49 min	97%	3%

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62 min 97% 3%

(Retention time of caffeine)

Caffeine: 27.2 min

From each area % determined here, the corresponding wt% was determined based on the standard substance.

[0048]

## Example 1

Green tea leaves (steamed tea leaves from Sri Lanka; 100q) were evenly mixed with an 80% aqueous ethanol solution (36.5 g), and were moistened at 5°C for 15 hours. The moistened green tea leaves were charged in a semi-batch supercritical carbon dioxide extraction apparatus, and were treated with carbon dioxide which contained 2% of an 80% aqueous ethanol solution, at 30 MPa and 70°C for 6 hours. The amount of carbon dioxide used for the treatment was 14 kg. Subsequent to the treatment, the extraction residue was dried under reduced pressure at 40°C to obtain supercritical CO2-treated green tea leaves (92 g). The green tea leaves were extracted at 92°C for 1 hour with ion-exchanged water (10 L), followed by filtration to obtain an extract. The extract was lyophilized to obtain a green tea extract (40 g). The contents of non-polymer catechins and caffeine in the extract were 48 wt% and 2.2 wt%, respectively, and the non-polymer catechins/caffeine weight ratio was 22.

The thus-obtained green tea extract (20 g) was suspended

in a 95% aqueous ethanol solution (98.18 g) at room temperature under stirring at 250 rpm. After addition of activated carbon ("KURARAY COAL GLC", product of Kuraray Chemical K.K.; 4 g) and acid clay ("MIZUKA ACE #600", product of Mizusawa Chemical Industries, Ltd.; 20q), stirring was continued for about 10 minutes. After a 40% aqueous ethanol solution (82 g) was added dropwise over 10 minutes, stirring was continued for 40 minutes at room temperature. After the activated carbon and precipitates were filtered out by No. 2 filter paper, re-filtration was conducted through a 0.2-µm membrane filter. Finally, ion-exchanged water (40 g) was added to the filtrate, and ethanol was distilled away at  $40^{\circ}$ C and  $3.4 \times 10^{-3}$  kgf/cm<sup>2</sup> to obtain a product. The contents of non-polymer catechins and caffeine in the product were 263 mg/100 mL and 4.3 mg/100 mL, respectively, and the non-polymer catechins/caffeine weight ratio was 61.

[0049]

# Comparative Example 1

Green tea leaves (steamed tea leaves from Sri Lanka; 100g), which had not been subjected to a treatment with supercritical carbon dioxide, were extracted at 92°C for 1 hour with ion-exchanged water (10 L), followed by filtration to obtain an extract. The extract was lyophilized to obtain an extract (44 g). The contents of non-polymer catechins and caffeine in the extract were 45 wt% and 7.5 wt%, respectively,

and the non-polymer catechins/caffeine weight ratio was 6.

The thus-obtained green tea extract (20 g) was suspended in a 95% aqueous ethanol solution (98.18 g) at room temperature under stirring at 250 rpm. After addition of activated carbon ("KURARAY COAL GLC", product of Kuraray Chemical K.K.; 4 g) and acid clay ("MIZUKA ACE #600", product of Mizusawa Chemical Industries, Ltd.; 20g), stirring was continued for about 10 minutes. After a 40% aqueous ethanol solution (82 g) was added dropwise over 10 minutes, stirring was continued for 40 minutes at room temperature. After the activated carbon and precipitates were filtered out by No. 2 filter paper, re-filtration was conducted through a 0.2-µm membrane filter. Finally, ion-exchanged water (40 g) was added to the filtrate, and ethanol was distilled away at  $40^{\circ}$ C and  $3.4 \times 10^{-3} \text{ kgf/cm}^2$ to obtain a product. The contents of non-polymer catechins and caffeine in the product were 247 mg/100 mL and 6.5 mg/100 mL, respectively, and the non-polymer catechins/caffeine weight ratio was 38.

[0020]

[Table 1]

		Example 10	Comp. Ex. 1
	Charged amount of green tea leaves (g)	100	100
step of green	Moistening solvent	80% aq. soln. of ethanol	
tea Leaves	Amount of moistening solvent (g)	36.5	
	Extraction pressure (MPa)	30	
	Extraction temperature $(^{\circ}C)$	70	
Supercritical	Extraction time (h)	9	
carbon dioxide treatment step	Added solvent	80% aq. soln. of ethanol	
	Amount of added solvent (wt%)	2	
	Amount of tealeaves recovered after treatment (g)	92	
	Extraction solvent	Ion-exchanged water	Ion-exchanged water
Extraction step for green tea extract	Amount of extraction solvent (L)	10	10
	Amount of extract (g)	40	44

[0051]

## Example 2

Using the tea extract obtained in Example 1 and the tea extract of Comparative Example 1, the citrus-flavored, packaged beverages described in Table 1 were produced, respectively. The thus-obtained beverages were taken by eight trained male assessors, and were assessed for the following five features. The results are shown in Table 2.

[0052]

### Assessed features

Tea flavor and taste (5-stage assessment):

- 1 Weak
- 2 Slightly weak
- 3 Average
- 4 Slightly strong
- 5 Strong

Citrus flavor and taste (5-stage assessment):

- 1 Weak
- 2 Slightly weak
- 3 Average
- 4 Slightly strong
- 5 Strong

Taste assessment (5-stage assessment):

- 1 Unpalatable
- 2 Slightly unpalatable

- 3 Average
- 4 Slightly tasty
- 5 Tasty

Changes in flavor and taste during high-temperature storage (3-stage assessment):

- A Not changed
- B Slightly changed
- C Changed

[0053]

[Table 2]

Example 2			(b)
	Added ingredients	Example	Comp. Ex. 1
	Conventional product*1	1	480
	Supercritically treated product*2	460	-
-	Artificial sweetener*3	8	8
	Sour seasoning	2.1	2.1
	Ascorbic acid	0.3	0.3
	NaCl	9.0	9.0
	KCl	4.0	0.4
	Dextrins	5	5
	Citrus fruit juice*4	2	2
	Citrus flavor*5	5.5	5.5
	Ion-exchanged water	Balance	Balance
	Total	1000	1000
ţ	Non-polymer catechins in beverage (wt%)	0.12	0.12
formulation	Non-polymer catechins/caffeine ratio in	61	38
	beverage (-)		
	Tea flavor and taste	П	4
	Citrus flavor and taste	5	3.5
Assessment	Taste assessment	5	2.5
results	Changes in flavor and taste during		
	high-temperature storage (products stored	Æ	บ
	at 37°C for 3 months)		

<sup>\*1</sup> Green tea extract of Comparative Example 1 \*2 Green tea extract of Example 10 \*3 Sucralose \*4 Grape fruit \*5 Grape fruit

# [0054]

It is appreciated from Table 2 that the packaged beverage according to the present invention, which used the extract from the tea leaves as the supercritical extraction residue, was extremely reduced in tea flavor and taste, exhibited the flavor and taste of the added citrus fruit juice and flavor, did not exhibit a green tea-derived flavor and taste which would have been otherwise developed after high-temperature storage, and was inhibited from flavor and taste changes.

[Document Name] ABSTRACT

[Abstract]

[Problem] To provide a packaged beverage, which contains catechins at high concentration, is lowered in green tea favor, and undergoes no change in flavor during storage at high temperatures.

[Solution] A packaged beverage containing from 0.06 to 0.5 wt% of non-polymer catechins, wherein the packaged beverage has been obtained by incorporating a tea extract obtained from tea leaves of the genus Camellia subjected beforehand to a treatment of contacting with carbon dioxide in a supercritical state.

[Chosen Drawing] None